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### CLAIMS

dispersion of ITO particles and the sheet silicate is parried out mautely. [Claim {]An interlayer, wherein it contains polyviny/racetal resin, a sheet silicate, a plasticizer, an adhesive strength regulator. ITO (tin dope indium oxide) particles, and a dispersing agent and uniform

dispersing agent 5.0 weight section, distributing and becoming further so that sheet silinates with a glass laminates according to claim 1 consisting of 0.1 to ITO particle 3.0 weight section, and 0.001 to one or more kinds of 0.0001 to metal sait 1.0 weight sections chosen from a group which consists of 20 to plasticizer 100 weight section, alkali metal salt, and alkaline earth metal salt. The interlayer for [Claim 2]Polyvinyl-acetal resin 100 weight section, 0.05 to sheet silicate 20 weight section, At least

size of 1 micrometers or more may be ten or less 100 micrometer2 hits.

of not less than 100 nm. The interlayer for glass laminates according to claim 1 or 2 currently [Claim 3]Mean particle diameter is 50 nm or less, and ITO particles in a lim are the particle numbers

distributing so that it may become one place less than I micrometer

silicate is an organicity-ized sheet silicate. [Claim 4] The interlayer for glass laminates according to any one of claims i to 3, whereir a sheet

[Claim 5] The interlayer for glass laminates according to any one of claims 1 to 4, wherein a dispersing recinoleic acid, and polyreninoleic acid. agent is at least one or more sorts chosen from a group which consists of a phosphoric ester system

acetsi resin is polyvinyl batyral resin. (Claim 6) The interlayer for glass laminates according to any one of claims 1 to 5, wherein polyvinyl-

(Otaim 7)A glass laminate using the interlayer for glass laminates according to any one of claims I to

less of visible light transmittance 1.0% or less. transmittance's being not leas than 70%, and solar transmittance (300 nm  $\sim 2500$  nm) being 80% or [Claim 8]The glass laminate according to claim 7 with which Hayes is characterized by visible light

[Translation done.]

JP,2003-201301,A [DETAILED DESCRIPTION]

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# DETAILED DESCRIPTION

[Detailed Description of the Invention]

[5000]

[Field of the Invention]This invention relates to the glass laminate which uses the interlayer for glass laminates and it which were excellent in transparency, thormal insulation nature, and electromagnation vave permeability, and adhesive strength with glass is sultable for, and were excellent also in panetration resistance.

(2000)

[Description of the Prior Art]Conventionally, since it is rare far the fragment of glass to disperse and it is safe even if it damages in response to an external shook, the glass laminate is widely used as windowpanes, such as a vehicle like a car, an airplane, and a building, etc. Generally what makes the intrologyer for glass laminates which consists of polyvinyl—acetal resin, such as polyvinyl butyral resin plasticized with the plasticizer, intervence between the glass of a couple at least as the abovementioned glass laminate, makes unity, and is obtained in used. The art of the interlayer is which the sheet silicate was minutely distributed in the film is also indicated by JP,2001—58853, A as an interlayer excellent in internal insulation nature to which importance was seldom attached until now as a glass leminate for a car or buildings on the other hand were mounting, withough the above conventional glass laminates were excellent in safety, they had the problem of being inferior to thermal insulation nature.

[0003]Generally, although an amount of energy is as small as about 10% compared with ultraviolet rays, since a thermal effect is large, and is absorbed by the substance and the infrared rays which have the wavelength of not less than 700 nm also in a beam of light bring about a rise in heat, they are called the heat ray. Therefore, in order to improve thermal insulation nature, the method of intercepting the windshield of a par, side glasses, and the infrared rays containing the glass window and glass door of a building is examised, and by for example, vocuum evaporation, sputtering processing, etc. The heat ray out glass etc. which provided coating layers, such as metal or a metallic exide, in the glass surface, and gave thermal insulation nature are marketed. This coating layer was weak from the oiltside to the abracion, and since chemical resistance was also inferior, the method of laminating interlayers, such as a plasticization polyvinyi-butyraf-rosin film, and using as a glass laminate, for example was adopted.

BDDM/However, the heat ray cut glass in which interlayers, such as the above-mentioned plasticization polyvinyi-butyral-resin film, were laminated. There was a problem that transparency (visible light transmittance) falls since it is expensive and the multilayer coating the stick, or the adhesive property of a multilayer coating the and an interlayer fell, and exhibition and the white blush mark of an interlayer happened. They ere various kinds of communication equipment (3.5 MHz), for example, a ham radio, in recent years. Although urgency traffic apparatus, (10 MHz or less), VICS (a car information communications system, 2.5 GHz), ETC (a full road electronic toll collection system, 5.8 GHz), satellite broadcasting (12 GHz), etc. are increasingly carried in a car, [ 7 MHz, ] The abovementioned multileyer coeting tip layer checked the penetration of electromagnetic waves, and lad problems, such as interfecing with communication functions, such as a cellular phane, car revigation, a garage opener, and a fee automatic acceptance system. A heat barrier is not provided in a glass surface, but the glass laminate which laminates is indicated by JP,61-52093.B. JP,64-35442.A public law.

http://www4.ipdi.inpit.go.jp/ogi-bin/tran\_web\_ogj.ejje?atw\_u=http%3A%2F%2Fwww4.ipdi.i. 2009/11/09

sto. However, there were problems, like exfoliation not only takes place, but as for a glass laminate the above-mentioned indication, there is a problem in the adhesive property between a plasticizati polyviryl-butyral-resin sheet and polyester film, and the interface of an electromagnetic wave penetration is also insufficient, and it is by it.

5000]

[Problem(s) to be Solved by the Invention]Intensity and pliability are compatible, and transparency especially Hayes are good, and the purpose of this invention is to provide the glass laminate using interlayer for glass laminates excellent in thermal insulation nature, and this interlayer for glass laminates, without moreover reducing electromagnetic wave permeability.

[9006]

iMeans for Solving the Problem]This invention contains polyvinyl-acetal resin, a sheet silicate, a plasticiter, an adhesive strength regulator, ITO (sin dope indium oxide) particles, and a dispersing agent, and ITO particles and a sheet silicate are the interlayers by which uniform dispersion is carout minutely.

[0007]In this invention, by using ITO particles, in order to raise thermal insulation nature, and carry out uniform dispersion of this ITO particle minutely in a film, transparency (especially Hayes) and electromagnetic wave permeability were able to be reduced, and thermal insulation nature was able to raisod without things. Intensity and pliability were able to be reconciled in this invention, without reducing transparency (especially Hayes), since a sheet silicate is used and uniform dispersion is minutely carried out into a film.

[0008]If polywhyl-acetal resin used by this invention is polyvinyl-acetal resin produced by acetaliz polyvinyl alochol (PVA) resin by aldehyde, it will not be limited in particular. The above-mentioned PVA resin is obtained by usually saponifying polyvinyl acetate, and, as for a saponification degree, 80-99.8-mclls of PVA resin is generally used. Although in particular a molecular weight and molecular weight distribution of polyacetal resin that are used for this invention are not restricted, from a moldebility, physical properties, etc., a thing of 200-3000 is preferably used for a degree of polymerization of FVA resin used as a raw material, and resin of the dagrees of polymerization of FVA resin used as a raw material, and resin of the dagrees of polymerization set and severage degree of polymerization resistance of a glass laminate obtained as the above-mentioned average degree of polymerization is less than 200 falls and the above-mentioned average degree of polymerization exceeds 3000, the moldability of a resin layer worsens, moreover the rigidity of a resin layer will become large too much, and processability will worsen.

20008]As long as are used, and a carbon number chooses suitably, and is used seconding to performance demanded, its \*\*\*\* is good and aldahyde of 1-10 is required for it as the above-mentioned addeliyde, two or more kinds may be used together. As an example of aldehyde, for example or-bucylaidehyde, isobutyraldehyde, and performance demanded in the control of aldehyde, removed aldehyde, isobutyraldehyde, in-alteriatelyde, 2-ethylbutylaidehyde, benzaldehyde, etc. are mentioned. As aldehyde, or-decyl aldehyde, isomaldehyde, astaldehyde, and nethylaidehyde, removed aldehyde, and nethylaidehyde, are mentioned. As aldehyde, or-decyl aldehyde, isobutyraldehyde, isobutyraldehyde, and nethylaidehyde are mentioned. A carbon number is butylaidehyde, is \*\*HEKI sill aldehyde, and netheraldehyde are mentioned. A carbon number is butylaidehyde of 4 especially preferably.

[1010]As desirable polyvinyl-acetal resin, polyvinyl-butyral (PVB) resin acetalized with butylaidehyd is mentioned especially. After these acetal resin bakes required physical properties into cansiderat it may be blended in a suitable combination. It is also possible \*\* polyvinyl-acetal resin which combined two or more kinds of aldehyde at the time of acetalization, and to use suitably. The degrated acetalization of said polyvinyl-acetal resin used by this invention is usually 40 to 85%, and is 60. 75% preferably.

[0011]A sheet silicate used by this invention has an exchangeable cation between layers of a deta flaky crystal about 1 nm thick, in [ are the silicate mineral condensed in layers by an ionic bond, ar this invention. The layer structure is exfoliated by a chemical or physical means, by distributing thi flake uniformly in a transparent resin composition, the transparency of a resin composition would thield and also a function as an inorganic bulking agent, a filler, and a viscosity controlling agent can exhibited in a resin composition.

[0012]Atthough e kind in particular of the above-mentioned shoot silicate is not limited, monumerillanite. There is a vermiculite, helloysite, or swelling mice besides smeetite system argilit such as saporite, hectorite, beidelitte, a stovensite, and nontronite, etc., and what was compounde [ a natural thing or ] can be used preferably.

[0013]As shape of a sheet silicate used for this invention, 0.01-3 misrometers and thickness are

JP,2003-261361,A [DETAILED DESCRIPTION]

ratio. more suitably, 0.05-2 micrometers and thickness are used for average length, and a thing of 50used for average laright, a thing of 20-500 is preferably used for 0.001-1 micrometer and an aspect 200 is used for 0.01-0.5 micrometer and an aspect ratio.

distearyldimethylbanzylasssonium salt, a Ji hardening beef tallow dimethylenomonium salt, distearyl earnest be strong, and cannot fully un-polarize between layers of a sheet silicate. As quarternary street silicate which it comes to process and which is not organicity-ized with a sation system easy to be distributed more minutely in resin than a sheet silicate as for which erganicity-reation is a silipate to become firm, and for INTAXARE by plasticizer and a cation system surface-active agent to dibenzylammonium salt, etc. are mentioned. trimethylammonium sait, a stearyl trimethyl AllMONIMU sail, trioctyl ammonium sait, a ammonium selt which has a with a oarbon numbers of eight or more altyl chain. For example, lauryl alkyl chain whose carbon number is eight or more, the hydrophilic nature of eikyl emmonium ion with a carbon numbers of eight or more alkyl chain preferably is used. When it does not contain an class phosphonium selt, etc. are meritioned, and quarternary emmonium selt which has at least one cetion system surface-active agant used far organicity-ization, quarternary ammonium selt, the 4th surface-active agent, the above-mentioned organisity-ized sheet silicato is used more suitably. As a organicity-rized sheet silicate. It is satisfactory in any way. Since between layers of a sheet silicate is Under the present circumstances, even if a sheet slicate which is not organicity-ized exists in an (0015) Although a sheet silicate may be used as it is and an organizity-ized sheat silicate organicitybecome insufficient, and to distribute a sheet silicate minutely, in exceeding 200 mmol/100 g. between crystal layers decreases easily, and a sheet silicate may not be distributed minutely as a plasticizer and a cation system surface-active agent which are intercalated by ionic exchange limited, it is preferred that it is 50 to 200 mms/100g, in a case below 50 mms/ts)/100g, quantity of a ization-processed beforchend may be used, it is preferred to use an organicity-ized sheet silicate. result. On the other hand, it may be difficult for associative strength between layers of a sheet 1991 4]Although cation exchange capacity in particular of a sheet silicate used for this invention is not

may be produced, and it is not desirable. An addition of a more desirable sheet silicate is 0.5 to 5 physical properties, such as a fall of transparency, aggravation of Hayes, and shock resistance, etc. weight sections, a pitch which stst occupies in an increase composite material may decrease, a fall of addition does not fully come to demonstrate desired physical properties but adds expending 20 polyvinyl-acetal resin 100 weight section. In less than 0.05 weight sections, when it is few, and an 10016]As for an addition of a sheet silicate, it is preferred that they are 0.05 to 20 weight sections to

pieces still more preferably. distribution. Quantity of a sheet silicate of 1 micrometers or more or an organicity-rized sheet silicate especially transparency that many sheet silicates with a viewing and a size of 1 micrometers or more which can be checked on a scanning electron microscope (SEM) level exist as a grade of the [0017]A sheet silicate needs to distribute minutely and it is not preferred on mechanical strength, is tan or less per 10 micrometera x 10 micrometers, and a desirable dispersion state is five or less

case, same plasticizers and the whole quantity of a sheet silicate are coce mixed, and a residue of a sheet silicate being mixed with resin becomes is easy to be distributed in resin minutely easily. In this a shaet silicate and a plasticizer beforehand, it is because the time of a plasticizer swelling and said swelled an interlayer spacing of a sheet silicate ix added and kneaded to resin is preferred. By mixing especially a thing for which a sheet slicate and a plasticizer are mixed beforehend, and what fully plasticizer may be applied further efter that and it may mix. [2018]Especially as a method of distributing a sheet silloate minutely in a film, although not limited

organic-acid exter system plasticizer, for example Triethylene glycol. Glycol and butancic acid, such glycol --\*\*\* orang acid ester, triethylane glycol di-2-ethylbutylate especially, Monobasic-organicheptylasid, n-octylis acid. Alycol system ester obtained by a reaction with monobasic organic acid, as tetraethylene glycol and tripropylene glycol, Isobutyric acid, caproic acid, 2-ethylbutanoic acid, nat limited, and phosphoric sold system plasticizers, such as organic acid ester system plasticizers, [0010]As a plasticizer, especially if conventionally used for an interlayer or polyvinyl-ecetal resin. It is such as 2-ethylhexyl acid, pelargonic acid (n-nonylic acid), and decylacid, is mentioned, Triethylene system, and an organic phosphorous acid system, etc. are used, for example. As a munchasiosuch as monobasic-urgsnic-soid exter and polybasic organio acid ester, an organic phosphorus acid

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> and penetration resistance may fall. When a plasticizer is added exceeding 100 weight sections, blooming the content of the co weight section. Less than 20 weight sections are insufficient for distributing a sheet slicate minut prong acid ester, triethylene glycol di-2-ethylbutylate, triethylane glycol di-n-notyl ecid ester, mentioned, Especially, dibutyl sebsolo soid exter, dicotylazelate, dibutylcarbitol adipate, etc. are us out of a plasticizer arises and there is a possibility that uptical strain of a glass laminate obtained in consideration of compatibility with resin, etc. according to a kind of polyvinyl-racetal resin. triethylene glycol di~2~ethylhexyl ecid ester, etc. are mentioned. These plasticizers are properly us (0020) As an example of a plasticizer used especially preferably. For example, triethylene glycol -\*\* suitably. As an organic phosphorus acid system plasticizer, tributaxyethyl phosphate, isodacyl pher ecid, and azelalo ecid, and the carbon numbers 4-8 or ester with letter alcohol of branching is desirable plasticizer is 30 to 60 weight section. the transparency of a resin layer and an adhesive property falling may become large. An addition o [0021]An addition of a plasticizer has 20 to 100 preferred weight section to polyvinyl-acetal resin \*\*\*\* of the above~mentioned plasticizer is also good, and two or more kinds may be used togetho phosphate, trisopropy) phosphate, atc. are mentioned, for example. One kind is used independently plasticizer, For example, straight chain shape of polybasic organic acid, such as adjnic acid, sebact glycol di-2-ethylhexyl acid ester, is used suitably. As a polybasic crysnic acid ester system acid ester of triethylane glycois, such as triethylene glyppi di-n-octyl acid ester and triethylene

meet and becomes an interlayer for glass from alkali motal selt and alkaling earth metal sait as an adhesive strength regulator is used. Especially as the above-mentioned metal, it is not limited, for carbon numbers 2-16 is used suitably aspecially. As still more desirable matel sait, they are magnesium acetate, potassium acetate, magnesium propionate, Potassium propionate and magnesi acetic acid, and formic acid, or chloride, and nitric acid, is mentioned, and a selt of organic acid of potassium, atc. may be used preferably, and these mey be used independently, or two or more sort numbers 2-12, or carboxylic acid potassium sait of the carbon numbers 2-12, For example, carboxylic acid magnesium salt of the carbon numbers 2-16, or carboxylic soid potassium salt of the mentioned salt, inorganio adid, such as organio acid, such as octylio acid, hexylacid, butanoic acid, example, sodium, potassium, magnesium, etc. are mentioned. As acid which constitutes the above-[0022]At least one or more kinds of metal salt chosen from a group of this invention which makes may be used together. 2-ethylbutanoata and potassium 2-ethylbutanoate, 2-hexenoic acid magnesium, 2-ethylbexanoic a varbon numbers 2–16. It is not limited especially as carbaxylic acid magnesium salt of the carbon

weight section, and an addition of at least one or more kinds of metal salt chosen from a group wh consists of the above-mentioned sikali metal salt and alkaline earth metal salt is 0.01 to 0.2 weight [0024]In this invention, in order to give thermal insulation nature, ITO particles are used. It is not limited that what is necessary is [ especially ] just the ITO particles which dope tin to indium axidations. under a high humidity atmosphere, and if 1.0 weight sections are exceeded, adhesive strength will and are usually used for addition of conductivity as ITO particles. As particle diameter of ITO section. In less than 0.0001 weight sections, an edhosive strength fall of a periphery takes place [3023]3.0001 to 1.0 weight section is desirable still more preferred to polyvinyl-acetal resin 100 100 nm, traosparency may falt. particles, 100 nm or less has proferred mean particle diameter of a primary particle. When it excee become law too much, and also a problem that membranous transparency falls will arise.

weight section. Content becomes difficult to come out of an infrared cutting effect in less than 0.1 weight sections, thermal insulation nature will fall, if 3.0 weight sections are exceeded conversely, i [3025]Centent of ITO particles has 0.1 to 3.0 preferred weight section to polyviny/racetal resin 10: permeability of visible light will fall, and Hayes will also become large.

will fall it is preferred to distribute that mean particle diameter is 80 nm or less, and a particle and when an ITO particle kind is not distributing uniformly minutely, transparency (especially Hayes [0026]It is necessary to parry out uniform dispersion of the ITO particles minutely into an interlays

number of not loss than 100 nm is below one piece/Imicrometer<sup>2</sup> as a dispersion state in inside of

in resin and uses, but in this invention, it is preferred to make it distribute as carrier fluid using a usually distribute uniformly in carrier fluid which consists of organic solvents, it is made to distribu [0027]It is not limited especially as a method of distributing ITO particles in resin, but after making

JP,2003-281381,A [DETAILED DESCRIPTION]

may be beforehend added by carrier fluid and a dispersing agent may be used for it, when mixing ITO into a lilm, and membranous Hayes can be made to improve further by using a dispersing agent. It [0028]In this invention, in order to carry out uniform dispersion of the ITO particles minutely into particles with resin, it may be added, and it may be used. resin, a dispersing agent is used Uniform dispersion of the ITO particles can be minutely carried out plasticizer used for an interlayer and a plasticizer of the same kind

acid are used suitably polyoxyethylene-alkyl-ether phosphoric acid, etc. are mentioned, for example, As a compound which has at least one or more carboxyl groupe, hydroxy acid especially recinoleic acid, and polyrecinoleic phosphoric ester system compound, sikyl phosphate, a polyoxysthylene alkylphonol ether phosphate. has at least one or more carboxyl groups are suifably used as a dispersing agent among these. As a more varboxy! groups, are mentioned. A phosphoric ester system compound and a compound which system compound, carboxylete, a polyhydric slockol type surfece—active agent, and at least one or particles, such as a compound which has a sulfate ester system compound, a phosphoric ester [0029]As a dispersing agent, dispersing agents generally used as a dispersing agent of inorganic

[0030]0.001 to 5.0 weight section is desirable still more preferred to polyvinyl-acetal resin 103 weigh may be improved too much. production and glass laminate creation, and may foam, or adhesive strength of an interlayer and glass expect but exceeds 5.0 weight sections, there is a possibility that it may be alike at the time of film quantity of a dispersing agent is less than 0.001 weight sections, and the addition effect can hardly section, and quantity of the above-mentioned dispersing agent is 0.005 to 3.0 weight section, when

and polyrecinaleic acid, Aliphatic carboxylio acid, aliphatic dicarboxylio acid, aromatic carboxylio acid, there is a possibility of foaming at the time of film production, or producing fearning at the time of and it is 0.01 to 1 weight section more preferably. When the amount part of duplexs is exceeded, agent, 0.001 - the amount part of duplexs are preferred to polyvinyl-acetal resin 100 weight section. condensation of ITO particles that Hayes improves. As an addition of the above-mentioned chelating methane, are desirable still more preferred elso especially in a cheleting agent, and an acetylacetone especially as the above-mentioned chelating agent, What has good compatibility with a plasticiter or dispersing agent, and a compound which has at least one or more carboxy/ groups except the abovescid, propionic axid, n-butancic scid, 2-ethybutsnoic scid, n-hexancic scid. 2-ethythexsnoic scid, nare used suitably, and are aliphatic carboxylic acid of C2~C10 more preferably. Specifically, acetio acid, etc. can specifically be used. Especially, aliphatic carboxylic acid of C2-C18 and hydroxy acid aramatic dicarboxylic acid, hydroxy acid, etc. are mentioned, and benzoic acid, phthalic acid, salicylic [0032]As a compaund with one or more carboxyl groups except the above-mentioned recincleic acid glass laminate production. An effect can hardly be expected to be less than 0.001 copy. is used suitably. When these chelating agents configurate to ITO particles, it is thought by barring resin is preferred, beta diketones, such as an acetylacetone, benzoyltrifluoroacetone, and dipivaloyi etc. are mentioned. Although it is possible not to limit and to use EDTA and hets—diketones mentioned recincleic acid and polyrecincleic soid as a distributed auxiliary agent, a cheleting agent, [003] JWhen distributing ITO partioles, a distributed euxiliary agent may be used in addition to a

adhesive strength of glass and a film, and the addition effect is hard to accept to be less than 0.001 weight section. resin 100 weight xection, and a more desirable addition is 0.01 to 1 weight scotion. When the amount recinateic acid and polyrecinoteic soid, it is 0.001 - the amount part of duplaxs to polyvinyf-acetal part of duplexs is exceeded, there are fear of membranous yellowing and a possibility of spoiling [0033]As a quantity of a compound with one or more earboxy! groups except the above-mentioned

octanuic soid, stc. ere mentioned.

needed in the range which does not check an effect of the invention. agent, and a fisat ray absorbent, may be added by interlayer for glass laminates of this invention if spray for preventing static electricity, an adhesive strength regulator, \*\*\*\*\*, a heat ray reflective [0034]Additive agents, such as an antioxidant, an ultraviolet ray absorbent, lubricant, fire retardant, a

Improvement in penetration resistance, etc. may laminate and use the interlayers for glass laminates weatherability are taken into consideration, it is proferred that it is 0.3 -- 0.8 mm practical. is limited — as a glass laminate — the minimum — when required penetration resistance and (0035)aithough thickness in particular of an interlayer for glass laminates of this invention is not what

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of this invention or an interlayer for glass laminates of this invention, and other interlayers if needs

feminate, what is called organic glass, such as polycarbonate, polymethylmethacrylate, etc. which rays can be intercepted and the thermal insulation effect can be heightened. As glass used as a gl heat absorbing glass whose transmissivity in a full wave laugth field (900 nm - 1300 nm) is 65% or which intercepts infrared rays of a wavelength erea (900 nm - 1300 nm) is preferred, and especiali currently generally used can be used. Although usual glass may be used as glass, heat absorbing gl were excellent in transparency in addition to inorganic glass, may be used with glass which ebsorbs infrared reys of a wavelength band (800 nm - 1300 nm), wide range infrar less is preferred. Since infrared outoff performance of ITO particles is large at the long wavelength for glass laminates, end glass, especially as glass, it is not limited but transparent plate glass side and comparatively smaller than 1300 nm in a wavelength ares (900 nm - 1300 nm). By combin [8036]Although obtained by an interlayer of this invention laminating the above-mentioned interlay

because it becomes. In this case, some plasticizers and the whole quantity of a sheet silicate are adhesive strength modifier. ITO particles, and a dispensing agent, and just to produce a film. Under \*\* elso of the ITO particles was carried out, adding, after carrier fluid has distributed beforehand is once mixed, and a residue of a plasticizer may be applied further after that and it may mix. As poin silicate spreads and it mixes with resin, easily, into resin, it distributes minutely and burns --- it is plasticizer beforehand, when a plasticizer swells in said sheet silicate, between leyers of a sheet distributed in a plasticizer is added and kneaded to resin is preferred, by mixing a shect cilicate an the present circumstances, especially thing of a sheet silicate for which what was beforehand glass laminates of this invention, to knead polyviny!-acetal resin, a speet silicate, a plasticizer, an [9037]What is necessary is not to be limited especially as a method of obtaining en interlayer for

invention but what is necessary is just to produce a film by extrusion process, the calendar metho Although it is not limited especially as a method of fabricating an interlayer for glass laminates of t is preferred to use an extrusion machine from a viewpoint of producing continuously especially. mechanochemicel device, a Henschel mixer, a homogenizer, en ultresonic irradiation machine, etc. plasticizer, and a dispersing agent is not limited, plenet type agitating equipment, a wet direction, and fleyos can be made to improve further. the pressing method, etc., it can be more preferably based on an extrusion process by the blaxial a an extrusion machine, plastograph, a kneader, a Banbury mixer, a calendering roll, etc. can be used plasticizer, an adhesive strength modifier. ITO particles, and a dispersing agent is not limited, eithe generally used. Although a device used for kneading of polyvinyl-acetal resin, a sheet silicate, a [8838]Although a device in perticular that mixes a plasticizer, a sheet silicate and ITO perticles, e

transmittance are not less than 10%, and Hayes is [solar transmittance] 80% or less of visible light transparency, and it solar trensmittence (300-2500 nm) is 80% or less of visible light transmittence [0030] As for a glass laminate of this invention, it is preferred that 1.0% or less and visible light by the side of long wavelength decreased rather than visible light. has the thermal insulation nature which was excellent since transmissivity of light of an infrared an less than 70%, it comes out and there is Hayes of a glass laminate at 1.0% or loss, it excels in transmitted light is a light whose wavelength is 300–2500 nm here, if visible light transmittance is r transmittance. Visible light refors to light whose wavelength is 380-780 nm, and the solar radiation

and a train, structural glass, etc. It laminates with other films and it is also possible by using to use conveniently for a glass part of vehicles, such as a windshield of a car and side glasses, an airpland etc. An interlayer for glass faminates of this invention is laminated with rigid bodies other than glas for example, as functional glass laminates, such as an insulation glass laminate which gave insulation [8040]A glass laminate using an interlayer for glass laminates of this invention can be used for example, metal, an inorganic material, etc., and application as a damping raw material is also

interlayer like a sheet silicate of buik, with an additive agent, visible light is scattered about strongl invades betwaan layers whan distributing a sheet silicate in rasin as mentioned above. Since is possible in this invention to distribute a sheet silicate minutely efficiently when a plasticizer and decline in visible light transmittance, aggravation of Hayes, etc. pose a problem. However, sino usually adds an additive agent of size beyond it to resin of transparency like an indispensable [D041](OPERATION) When equivalent to a visible light wavelength or high visible light transmittanc

JP,2003-261361,A [DETAILED DESCRIPTION]

transparency is secured and it is not necessary to remove a plasticizer, an interlayer for glass laminates of outstanding physical properties is obtained easily. That is, an interlayer for glass laminates which is the purpose of original inorgenic substance addition and which retining (coexistence of mechanical-strength intensity and plability) of an interlayer of became possible, and was excellent in transparency, and reconciled mechanical-strength intensity and pliability is obtained with high visible light transmittance meintained.

[0942]On the other hand, it is transparent, and an interlayer with the thermal insuiation effect is obtained, holding transparency, since ITO perticles which moreover have infrared absorption ability were minutely distributed by interlayer. Since ITO perticles are smaller than visible light enough since it distributes uniformly in the state of a nation-scale ultraffine particle, and dispersion is not exused an interlayer excellent in transparency, especially a heze value is obtained. Since ITO particles are minutely distributed in an interlayer with a gestalt of an ultraffine particle. It differs from \*\*\*\* glass and a heat ray reflective glass laminate using heat ray reflective PET by vacuum evaporation or coating which were conventionally used for a thermal insulation nature interlayer. When reflection of a communication functions, such as a callular phone, car navigation, and a garage opener, it does not become a problem at all. By distributing a pissticizer beforehand and furthermore, using an ITO ultraffine particle, it can process like a manufacturing method of the usual interlayer, processability, workability, preductivity, etc. are not spoiled, and it is obtained as usual.

[0.043]The more a sheet silicate generally distributes a sheet silicate manutely in resin as a secondary effect with addition sweet red bean soup with modin, the move a machanical strength of a thermoplastics—sheet silicate composite, gas barrier property, and transparency improve remarkably. When an interface product of a sheet silicate and resin increases with improvement in distribution of a sheet silicate can explain. That is, polymor intensity can be efficiently increased, so that a distributed degree of a sheet silicate improves, since dynamics intensity, such as an elastic modulus of polymor, increases by restraining molecular motion of polymor in an interface of resin and an inorganic erystal. Since a reain layer tends to diffuse a gas molecular far as compared with an inorganic substance, when a gas molecular officiently maked, so that a distributed degree of a sheet silicate improves. By composite material, it is spread bypassing an inorganic substance. Therefore, a gas barrier can be efficiently maked, so that a distributed degree of a sheet silicate improves. By composite—izing an ITO ultrafine particle and a sheet silicate with a nano-scale as mentioned above, it becomes possible to give various functions to an interlayer.

[Example]Hereafter, the contents of this invention are explained based on an example and a comparative example.

Exemples 1-9, the comparative examples 1-4, 6-7 [Composition of a polyvinyl butyral] The heating and dissalving of 275 g of the PVA resin (sverage-degree-of-polymerization 1700 and saponification degree % of 99.2 mol) were added and carried out to the pure water 2890g. Temperature control of the system of reaction was carried out to 15 \*\*, 35% of the weight of the chloride 281g and the n-butylaidehyde 157g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 60 \*\* for 3 hours, and the reaction was made to complete, superfluous water washed unresacted n-batylaidehyde was flushed, sodium bydroxide solution neutralized the chloride catalyst, and white powdered PVB resin was obtained through rinsing and desicocation for 2 hours with still more superfluous water. The degree of formation of average butyral of this resin was 68.5-mol %.

[0045][Production of an ITO distribution plasticizer] As opposed to triethylene glycol di-2-ethyl HEKISHI rate 20 weight section, ITO powder (mean particle diameter of primary particle: 30 nm) 1 weight section was baught, and ITO particles were distributed in the plasticizer in the level type micro bead mill, using phosphoric ester of nonylphenyl polysthylene oxide as a dispersing agent. Then, it added under stirring of acetylacetone 0.1 weight section in the solution concerned, and the ITO distribution plasticizer was produced. The mean particle diameter of the ITO particles of a distributed electricizer was produced. The mean particle diameter of the ITO particles of a distributed electricizer was produced.

[Production of a sheet silicate distribution plasticizer] Triethylane glycoi di-2-ethyl HEKISHI rate 20 weight section and awelling mice (trade name MAE, GO-OP CHEMICAL GO, LTD, make, organicity-ized processing article) I weight section were mixed for I minute with planet type agitating

http://www4.ipdt.inpit.go.jp/cgi-bin/tran\_web\_cgt\_cjje?atw\_u=http%3A%2F%2Fwww4.ipdt.t.\_\_\_2003/11/08

equipment, and the paste state sheet silicate distribution plasticizer was obtained

[9048][Manufacture of the interlayer for glass laminates] PVB resin 100 weight section obtained above, the specified quantity of an ITO distribution plasticizer in which ITO particles become the quantity of Table 2. The specified quantity of a sheet silicate distribution plasticizer in which a she silicate becomes the quantity of Table 2. The specified quantity of the plasticizer that the total amount of a plasticizer (triethylone glycol di-2-ethyl HEKISH rate) becomes 40 weight sections. After fully carrying out melt kneading of the specified quantity of 2-ethylbutancic soid magnesium which magnesium content is furthermore set to 80 ppm to the whole system with a roll mill, press forming was carried out for 30 minutes at 150 \*\* using the preser-forming mechine, and the interla of 0.76 mm of average thickness was obtained. The mean particle diameter of the ITO particles in film is 50 nm, and as for a unt leas than 190-run particle, particle diameter was not observed. A sheet ellicate of 1 micrometers or more was not observed.

[0047][Manufacture of a glass laminate] The interlayer for glass laminates obtained above is put in the hoth ends with a transperent float glass (30 cm by 30 cmx2.5 mm in thickness), After putting this in the rubber back and carrying out indirect desulfurnation mind with the degree of vecuum of 20torr for 20 minutes, it moved to even, deaerated, and the vacuum press was carried out, holding 30 minutes at 90 more \*\*. Thus, sticking by pressure was performed for the glass laminate by which preparative pressure arrival was carried out for 20 minutes on condition of 135 \*\* and pressure 1.2MPn in autoclave, and the glass laminate was obtained.

[0048]It cerried out like Example 1 except using the ITO distribution plasticizer which made comparative example 5 triethylene-glycol di-2-ethyl HEKISHI rate 20 weight section distribute ITO particle 5 weight section.

It earried out like Example i except using the sheet silicate distribution plasticizer which made comparative example 8 triethylene-glycol dr-2-ethyl HEKISHI rate 20 weight section distribute she silicate 23 weight section.

The glass laminate was produced using the usual interlayer (average 0.78 mm in thicknose) which does not contain comparative example 91TO and a sheet silicate as heat reflective glass with which lamination coating of a silver film and the thin film of a metallic oxide was carried out in one sheet the float glass used when producing a glass laminate.

By two usual interlayers (average 0.38 mm in thicknoss) which do not contain comparative example 101TO and a sheat silicate, the silver film and the thin film of the metallic oxide put heat ray reflective PET by which lamination coating was corried out, inserted with the transparent float glanfrom both ends further, and created the glass laminate.

[0049][Evaluation] The following evaluation was performed about the interlayer for glass laminates end glass laminate which were obtained by the above—mentioned example and the comparative example, and the result was summarized in Tables 2 and 3.

 The transmissivity of 300~2500 nm of e gless leminate is measured using an optical property Na spectrophotometer (Shimadzu "UV3100"). It asked for the visible light transmittance (Tv) of 380-; ign, and the solar transmittance (Ts) of 300-2500 nm by JIS Z 8722 and JIS R3106 (1888).

2) Hayes of the glass laminate was messured based on Hayes dIS K 8714. [0050]3) The transmission electron microscope (TEM) was used after super-flake production of the glosopersion state interlayer in the inside of the film of ITO particles, and the dispersion state enterlayer in the inside of the film of ITO particles was made into the long particles was photoid and observed. The particle diameter of ITO particles was made into the long path of the ITO particles in the photograph obtained by the above-mentioned photography. The particle diameter of all the ITO particles in 10 micrometers × 10 micrometers of the above-mentio photographing areas was measured, and it asked for mean particle diameter with a volume convers average. It asked for the particulate number with a particle diameter of not less than 100 nm which exists in the above-mentioned photographing area, and the number of par 1 micrometer<sup>2</sup> was exists in the above-mentioned photographing area, and the number of par 1 micrometer<sup>2</sup>.

(1) A viewing device, a condition and a transmission electron microscope:H-7100FA type (made by Hitashi, Ltd.)

eccelerating voltage: -- 100-kV(2) section manufacturing device and ultramicrotome: -- ew-u.re/s (made by the Leica company)

Freezing cutting system: REICHERT-MISSEI-FCS (made by the Leica company)

Knife: DIATOME BLIRA CRYD DRY (made by DIATOME)

more, and asked for the number of per i micrometer? in arbitrary positions. 4) By making the interfayer for dispersion state glass laminates in the inside of the film of a sheet silicate into a sample, using the scanning electron microscope (SEM), the range of 10 micrometers x 10 micrometers was observed, and the mejor axis asked for the particle number of 1 micrometers or

(1) A condition and viewing device and scanning electron microscope:S-3500N (made by Hitachi, Ltd.)

6) The pan Meru value glass laminate was neglected to ~18\*\*0.6 \*\* temperature for 16 hours, and wave number range, it was considered as the electromagnetic wave shielding index. was measured in quest of the maximum and the minimum of a loss in the above-mentioned perimeter method of a distant place community), and the rate of reflection loss of the range of 2-26.5 GHz (6B) received the electric wave from a radio wave signal generator (electromagnetic wave measuring 600 mm square is stood among one pair for transmit receives of antennas. The spectrum analyzer measurement (electromagnatic wave shielding effect measurement of a neighborhood community) Measure the reflection has value (dB) of the range of 0.1-10 MHz, and, on the other hand, semple Accelerating voltage: 15 kV [0051]5) By the electromagnetic wave permeability KEG method

property over the glass of an interlayer is estimated by the pan Meru value, it is so large that a pan beforehand, and the pan Meru value was calculated for the result according to Table I. The adhesive out grade attachment of the degree of exposure of the film after glass carries out partial avulsion Mechanical strongth (tensile strength) Meru value is large, and adhesive strength is small when small. Lof adhesive strength with glass I particle diameter of glass was set to 0 mm or less. It judged with the boundary cample which carried was adjusted to it, and it ground until the head struck this with the hammer which is 0.45 kg and the

condition of temperature:23 \*\*, humidity:50%RH, and speed-of-testing:260 mm/min using the The high-speed hauling examination of the interlayer was done with the universal testing machine on dumbbell No. 1 type specimen (JIS-K-6771).

8) The humidity test constant temperature/humidity chamber was used and the humidity test of the glass jurninate was done based on JIS R 3212(1992)"automobile safety glass test-method." 0052

パンメル依 数6%托架(%) 000 ø 1C. ф: С77 ķ. œ O **(7**) 2 ٨ 8 b1 بىر دې ¢ ~ ŧ): 2 X 7 œ

[0053] [Table 2]

[0054] [Table 3]

### 35 × 阙 4 5 8 1 2 3 5 7 В 2.0 **密状总数均添加盈(食量剂)** 1.0 0.5 1.0 9.5 1.0 0.5 2.0 2.0 1.0 0.3 0.3 2.0 2.0 0.3 3.0 2.0 (新麗麗) 整成線の工( 1.0 0.02 マイカ分散性 1 μ加超粒子数 (競/μm²) 0.00 0.00 0.00 0.00 0.01 0.00 0.02 0.02 1TC 分数性 6.7 59 58 54 61 63 ß į s 1 6 8 平均数子等 (nm) 0.00 0.66 0.40 0.00 0.00 100m超粒子数(硬/4m\*) 0.00 0.00 0.00 0.50 76.8 可视光透透率 (%) 8 2. E 83.1 87.3 87.1 77.2 77,5 86.9 8 2 : 8 光学特性 日教養経帯(%) 66.2 55.8 63.2 62.7 47.0 47.5 62.2 85.2 47.1 0.6 0.8 ヘイズ (%) 0.5 0.4 0.4 0.5 0. 7 0.4 0.4 0~1 電磁波シールド性 (BdB) 0~1 0~1 0~1 0~1 0~1 0~1 0~1 9 -~ 1 引っ張り強度 (MPs) 38.7 39.9 34.8 40.1 34.5 42.9 43.1 43.5 34.0 数物性 0 0 0 O 数数数数数数数 O 0 0 0 0 6 5 5 ä 5 8 S ñ バンメル弦 5

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laminate using the interlayer can be provided. especially Hayes are good, and ] for glass lanimates which can adjust adhesive strength, and the g

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[Translation done.]

		<b>丝 較 领</b>									
		1	2	3	4	8	ń	7	8	9×17	16*
等行行政党系四条(联条系)		0.0	0.01	5, Ç	7.0	1.0	0.5	0.01	13.0	-	
(TO添加量 (重量部)		9.0	0.0	0.55	0.08	8.0	1.0	1,0	0.0	-	
マイカ分散性	1 μ加州拉子数(加/μμ/)		0.06	-	0.00	6.03		0.00	0.10		
1 TO分散性	平均粒子藻 (nm)	~	-	5 Q	58	103	5 7	5.8		-	
	100mm包包子数(概/µm²)	-	-	9.28	0,80	3.00	0.00	0.50			
光学物性	可能光透影率(%)	89.0	88.9	1.8E	87.8	66.2	88.6	82.8	60.4	80.0	78.0
	日射透過率(%)	80.2	80.2	73.4	72.7	31.5	55.0	\$ 5.1	58.2	47.0	47.0
	ヘイズ (%)	0.5	Ű. 5	0.5	0.5	3.3	0.5	8.5	3.9	6.6	0.7
<b>考察</b> なシールト性 (Δ 3 B)		0~1	0~1	3~1	0~1	0-1	8~1	0~1	0~1	15-48	13~37
Service.	引っ張り始度 (Mドa)	29.0	3.9.1	29.4	39.8	39.4	28.6	29.7	77.4	38.6	63.0
	<b>新提定款金融</b>	0	0	٥	٥	0	0	0	O	×	×***
	パンメル総	\$	5	8	ŝ	ş	ş	5	5	ē	5
*fix		×	×	×	×	×	×	×	κ.	×	%

[0055] [Effect of the Invention]According to this invention, by making polyvinyl-acetal resin distribute a sheet silicate minutely, Reconcile intensity and pliability, excel in transparency and thermal insulation nature by carrying out uniform dispersion of the ITO ultrafine particle into a film further, and moreover electromagnetic wave permeability is good. The interlayer [ it is cheap, and transparency,

http://www4.ipdl.inpit.go.jp/ogi-bin/tran\_web\_ogi\_cije?atw\_u=http%3A%2F%2Fwww4.ipdli... 2009/11/09

<sup>※21</sup> 熱級記針FBT使用 ※31 PST/中間線開網線